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ABSTRACT

The materials chosen for depth graded multilayer designs for hard x-ray telescopes (10 keV to 80 keV) have until now been focusing on W/Si, W/SiC, Pt/C, and Pt/SiC. These material combinations have been chosen because of good stability over time and low interface roughness. However both W and Pt have absorption edges in the interesting energy range from 70 - 80 keV. If looking at the optical constants Cu and Ni would be good alternative high-Z candidates since the k-absorption edges in Cu and Ni is below 10 keV. We have investigated both of these materials as the reflecting layer in combination with SiC as the spacer layer and give the performance in terms of roughness, minimum obtainable d-spacing and stability over time as deposited in our planar magnetron sputtering facility. Likewise we review the same properties of WC/SiC coatings which we have previously developed and which allow for very small d-spacings. The combination of WC/SiC or the well established W/SiC with the above mentioned Cu and Ni-containing multilayers in the same stack allows for novel telescope designs operating up to and above 100 keV without the absorption edge structure.

Keywords: Hard X-ray multilayer, stacked multilayer

1. INTRODUCTION

Soft X-ray telescopes like Chandra, ROSAT, and XMM are working at energies up to 10 keV use external reflection from Ir or Au coatings in a Wolter I geometry. For higher energies the critical angle is small, the angle below which there is total external reflection, so the high energy response for the above mentioned telescopes are very small. Since depth graded multilayer coatings were suggested¹ as a way to make focusing hard X-ray telescopes above 10 keV there have been done an extensive work in understanding and optimizing coatings for such telescopes. There have been flown two balloon payloads with conical approximation to a Wolter I optic coated with depth graded multilayer to demonstrate the concept. In 2004 the International Focusing Optics Collaboration (InFOCUS)² flew a single replicated mirror optic with a outer diameter of 400 mm, deposited with Pt/C multilayers and optimized for 20 keV to 40 keV. In 2005 the High Energy Focusing Telescope (HEFT)³ flew three slumped glass optics each 240 mm in outer diameter, deposited with W/Si and optimized for 20 keV to 69 keV. Several satellite missions whose main goal are the energy band above 10 keV and below 100 keV are currently planned, and all intend to use multilayers such as NASA's NuSTAR⁴ and Constellation-X⁵, ESA's XEUS⁶ and Japans NeXT⁷. Also the GRI mission are consider to incorporate a multilayer coated telescope to cover the energy range from 20 keV to 200 keV⁸.

The Danish National Space Center, DNSC, has for a long time been involved in the development of multilayers for hard X-ray optics, and besides having coated the three HEFT optics, DNSC will also be coating the optics for NuSTAR, and are producing test coatings for XEUS and other future hard X-ray and soft gamma-ray missions. Thus far W and Pt based coatings has reigned supreme in the hard X-ray energy band because of its relative low interfacial roughness and excellent stability over time, however, W has an absorption edge at 69.5 keV and Pt has one at 78.4 keV which is very close to the two ⁴⁴Ti lines⁹ at 67.9 keV and 78.4 keV. ⁴⁴Ti is very important to measure and since ⁴⁴Ti is produced at the boundary of the exploding envelope of a super novae explosion and can therefore be used to trace any asymmetry in the explosion. The light curves from super novae explosions are well understood, however, the explosion mechanism is not.

We will in this paper first describe the coating facility and then discuss some different material combinations that could be relevant for hard X-ray telescopes. Then we will show an example of a stacked multilayer coatings build up of two



Fig. 1. The coating facility at DNSC with four cathodes. The cathode in the front and to the right is in the “open” position, ready to sputter where the one to the left and the one in the back are in “close” position. The samples are mounted on mounting plates placed on the big ring in the bottom, the front half of the mounting plates have been removed. There are 16 mounting plates and two dummy plates on the ring. The targets in the cathodes are 500 mm tall, 38 mm wide, and the ring is a little over one meter in diameter.

multilayers with different material combinations, one on top of the other, and shortly discuss the benefit of such a stacked coating for future hard X-ray missions.

2. COATINGS

Since the coating of the HEFT telescopes the facility at DNSC have been modified so it now contains four DC magnetron sputtering cathodes with target size of 500 mm tall and 38 mm wide, see fig. 1. By masking along the cathode with an experimental found mask there is a 300 mm area on the cathodes that gives a constant coating rate within $\pm 2\%$ ¹⁰. The cathodes are sputtering outwards and shutters can be moved in front of the cathodes. Going from two to four cathodes have two purposes, first the possibility to coat the same material in two cathodes at the same time and that way increase the coating rate and decrease the coating time for long coatings. The other reason is the possibility to make coatings using more than two materials, like interface engineering, and as we will describe in this paper to make two different coatings on top of each other.

The substrates are mounted on mounting plates that are placed on a sample ring that can rotate. There are 16 sample mounting plates and two dummy plates on the ring. When the coating parameters are set, the thickness of the coating is only a function of the speed by which the substrate is moved by the cathode. By placing samples on every second sample mounting plates and use the empty mounting plates for acceleration it is possible to make 8 different constant d-

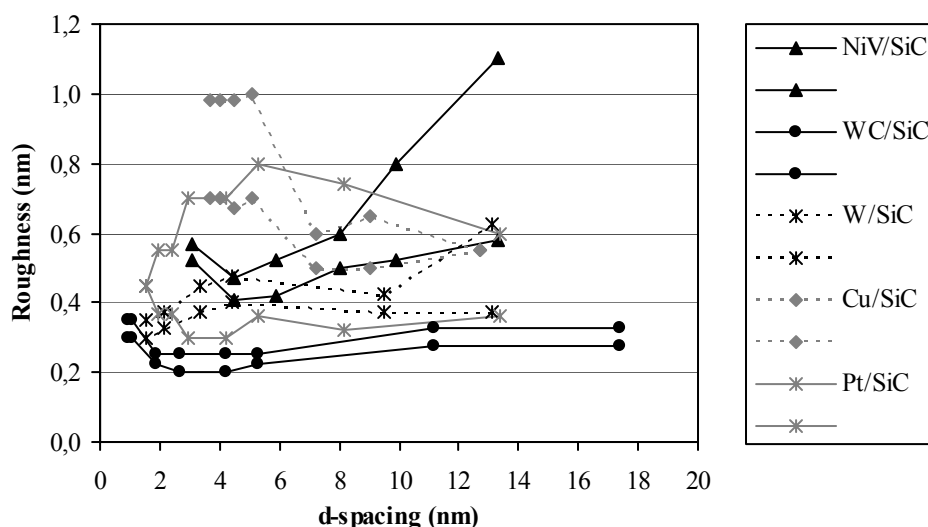


Fig. 2. Roughness as function of d-spacing for different high Z-materials combined with SiC as separator layer. For each material combination there are two lines, one for the roughness for A on B, and one for roughness for B on A.

spacing coatings in one run. The target-sample distance are 105 mm and the substrate are placed between two separator plates that have the width of the mounting plates and reach 30 mm into the chamber towards the cathode. The distance between the separator plates are 60 mm and they are used for collimating the coated material.

The following materials have been sputtered for this study, the number in the parenthesis is the purity: Cu (99.999%), Si (99.999%), SiC (99.5%), $\text{Ni}_{0.93}\text{V}_{0.07}$ (99.95%), Pt (99.99%), W (99.95%), WC (99.5%). The Si is doped with 32 ppm B and the SiC is doped with 6900 ppm B. Since pure Ni is magnetic we use an alloy with 7% V which make it nonmagnetic. For the rest of the article we will use NiV instead of the more correct $\text{Ni}_{0.93}\text{V}_{0.07}$. The substrates are commercially available Si wafers with a roughness of 0.25 nm measured with 8 keV X-ray. The sputter gas were Ar, purity 99.9999%, at 0.4 Pa (3 mTorr) and the coating are started when the pressure in the chamber was below 2.6×10^{-4} Pa (2×10^{-6} Torr). All X-ray data presented are made in house using a rotating anode 8 keV (Cu k_{α}) and the data analysis is made using IMD¹¹. We have used the optical constants for amorphous Si and SiC for the fitting. If nothing else are mentioned all samples are 30 bilayer constant d-spacing samples with a gamma, the high Z-material fraction of the total bilayer thickness, between 0.35 and 0.48.

3. MATERIAL COMBINATIONS

The standard way to fit X-ray data from constant d-spacing multilayers is to fit a d-spacing, a gamma, and a roughness of the interfaces. For some material combinations this give good fit but for other, or if a more detailed fit is desired, one need to use different interface roughness value for the two different interfaces in the coating to get good fit to the data, A on B and B on A. From the reflectivity data it can be hard to distinguish between which interface correspond to which roughness so in this paper we will give two roughness values, one for each material interface but not try to determine which roughness corresponding to which interface. The average of the two roughness values is close to the roughness value one gets by using one roughness for the fitting if the two roughness values are close. If the two roughness values are far apart it is not possible to get a good fit to the data with one roughness. When working with many layers of depth grating multilayer the average value works fine for fitting the roughness.

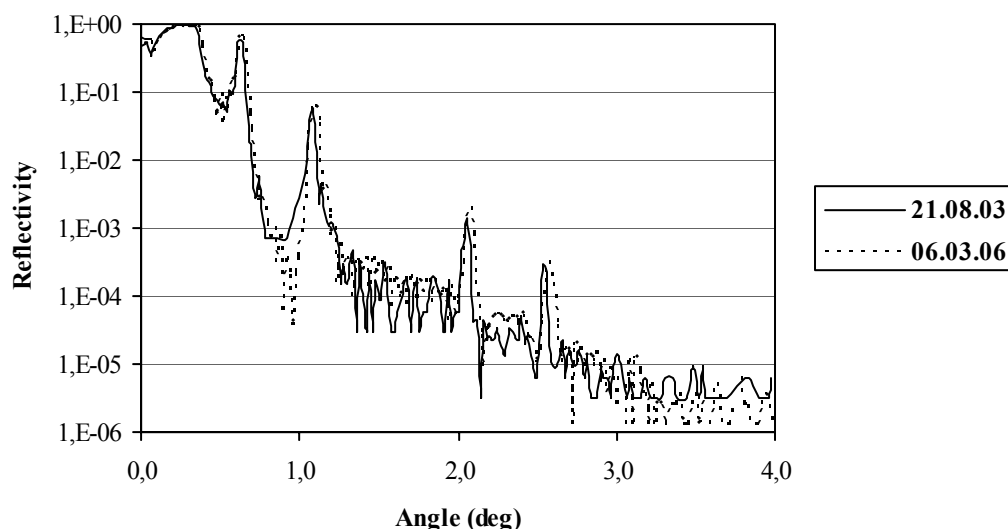


Fig. 3. A 10 bilayer Cu/SiC coating with a d-spacing of 8.7 nm, gamma of 0.67, and a roughness of 0.50 nm. This film was measured just after being coated and then 2½ year later.

The roughness of the interfaces has to be small compared with the d-spacing of the coating. For two reflection grating incidence hard X-ray telescopes the experience gives that the roughness divided with the d-spacing have to be below 1/6. This gives the lower level for how thin a coating of a specific material combination that can be used.

Comparing the traditionally used W/Si and with W/SiC, W/SiC, and WC/SiC coatings it is seen that the roughness decrease when using SiC instead of Si in combination with both W and WC¹². For these material combinations there is only a small difference between the two roughness values, below 0.1 nm. It is worth to note that WC has a 22% lower density than W.

Just looking at the optical constants Cu and NiV are more interesting materials for hard X-ray telescopes than W and Pt since both W and Pt have absorptions edges in this energy range. Both Cu and NiV have lower electron density than W and Pt but since the absorption is lower one can add more layers to the coating. The problem with Cu and NiV is that they are coating much rougher than W. It is therefore not possible to make as thin coatings with Cu and NiV. In fig 2 can be seen that WC/SiC have a much lower roughness than NiV/SiC, Cu/SiC, W/Si, and Pt/SiC. Because of the higher roughness, the smallest useful d-spacing that can be used for Cu/SiC is around 5.0 nm and for NiV/SiC it is around 3.7 nm. As for W and WC we have seen that Cu and NiV coatings have lower interface roughness with SiC than with Si.

It has earlier been reported that Cu/Si were stable at room temperature over a period of several months but showed visible degradation after 10 to 20 months¹³. Our Cu/Si coatings are still only 3 months old and show no degradation effect either optically or with X-ray. We have studied Cu/SiC for a longer time and as fig. 3 shows there is no degradation of the film after 2½ year. We have no explanation for this difference between Cu/Si and Cu/SiC but expect a build up of a C rich layer at the interfaces that works as an inter-diffusion barrier.

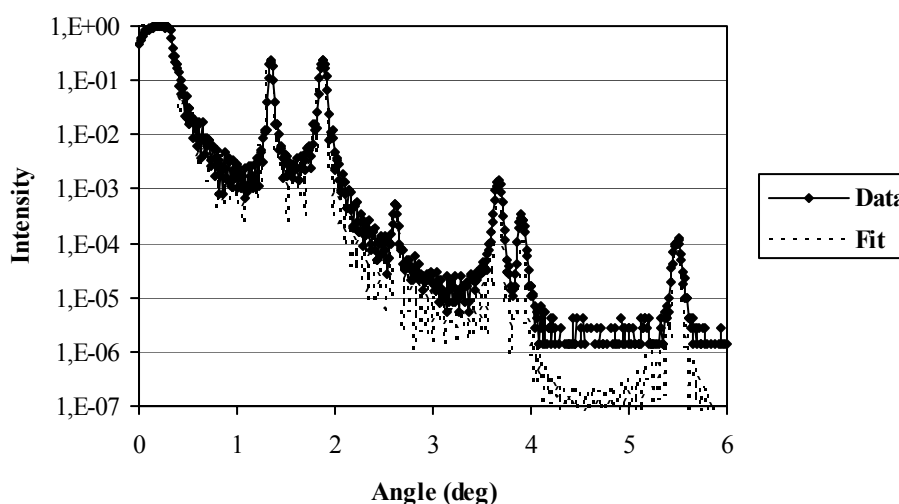


Fig. 4. A 30 bilayer constant d-spacing NiV/SiC coating on top of a 30 bilayer constant d-spacing WC/SiC coating on a Si wafer. The fit parameters can be found in table 1.

	d-spacing (nm)	Gamma	Roughness (nm)
NiV/SiC	3.38	0.45	0.45
WC/SiC	2.41	0.44	0.26
Si (substrate)			0.25

Table 1. The fit parameters for the data in fig. 4.

4. [NiV/SiC]/[WC/SiC]/Si multilayer

When a focal length and a energy range for a telescope have been chosen the minimum d-spacing can be calculated from Bragg's law. The focus have therefore until now been on material combinations that could be made with small enough d-spacings and materials like Cu and NiV have therefore not been used. Some telescope designs have different coatings on the outer and inner mirrors of the nested optic to optimize the throughput of the telescope. The above section describe some different material combinations and their advantages and disadvantages. Instead of chousing one material combination all the way down through the coating one could use the material combination with the best optical properties until the roughness is 1/6 of the d-spacing and then use other material combination to make the thin layers in the bottom of the coating.

In fig. 4 are shown the data for a coating of 30 bilayer constant d-spacing NiV/SiC on top of 30 bilayer constant d-spacing WC/SiC coating on a Si wafer. The fit parameters are shown in table 1. To simplify the fit there is only one roughness for each material combination. The found roughness values are in good agreement with the average roughness for that specific d-spacing for the two materials, compare with fig. 2. The d-spacing of the two material combinations is chosen to give a clear difference between the Bragg peaks coming from the different parts of the two different d-spacings, so Bragg peak 1, 3, and 5 relates to NiV/SiC and Bragg peak 2, 4, and 6 relates to WC/SiC.

In fig. 5 are shown a reflectivity curve of a multilayer with 100 bilayer NiV/SiC power-law depth graded¹⁴ coating on top of a 70 bilayer linear graded WC/SiC coating on a Si substrate. The design parameters are shown in table 2. From

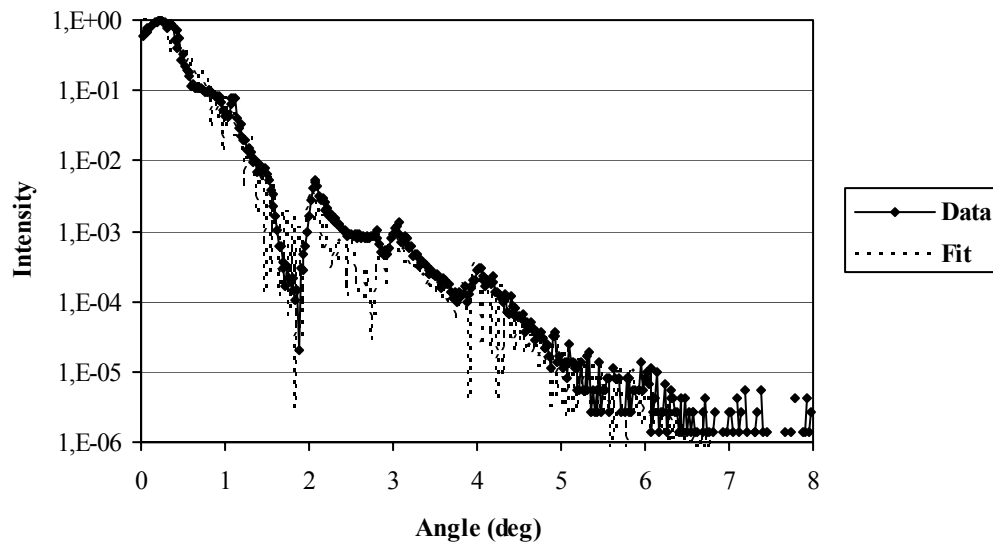


Fig. 5. A 100 bilayer power-law graded NiV/SiC on top of a 70 bilayer linear graded WC/SiC coating on Si. The parameters for the coating design and the fit parameters are shown in table 2.

	Design parameters					Fitted values
	d_{\min} (nm)	d_{\max} (nm)	N	c	Gamma	Roughness (nm)
NiV/SiC	4.6	26.0	100	0.28	0.45	0.58
WC/SiC	3.0	4.6	70		0.40	0.22
Si (substrate)						0.25

Table 2. The parameters for the coating design and the fit parameters for the calculated curve in fig. 5. The only fitted parameters are the roughness.

fig. 2 we know that the roughness is a function of the d-spacing so in principle one should build up the exact model of the coating with 170 different bilayer thicknesses, 170 different gamma values (there is a small dependency between gamma and d-spacing), and two times 170 different roughness values. Instead of that we use the assumption that gamma is constant and only fit one roughness parameter to the NiV/SiC coating and one to WC/SiC and then use the design parameters. This gives that the roughness for the NiV/SiC is 0.58 nm and for WC/SiC is 0.22 nm. These values seems to be in good agreement with the curves at fig. 2. The total thickness of the coating is 940 nm (670 nm NiV/SiC and 270 nm WC/SiC) and the stress of the coating is measured to -142 Mpa¹⁵.

By stacking two different depth graded multilayers on top of each other one can benefit from the properties from both. This gives a lot of new parameters when designing telescopes. One can increase the throughput at some energies and reduce throughput at other giving a more flat response curve and/or one can increase the maximum energy for the telescope. Fig. 6 shows a optimized power-law depth graded coating for the NuSTAR telescope calculated for an angle of 0.11 deg. The other curve is an example of a curve using two stacked power-law depth graded NiV/SiC and WC/SiC coating at the same angle. All the parameters for the two curves are listed in table 3. The main difference between the two curves is that the W/Si coating have a higher throughput between 30 keV and 43 keV but when the absorption edge of W kills the throughput at 69.5 keV there is still throughput using the stacked coating. For a full discussion of the impact these kinds of coatings have on future telescope designs please read reference 16.

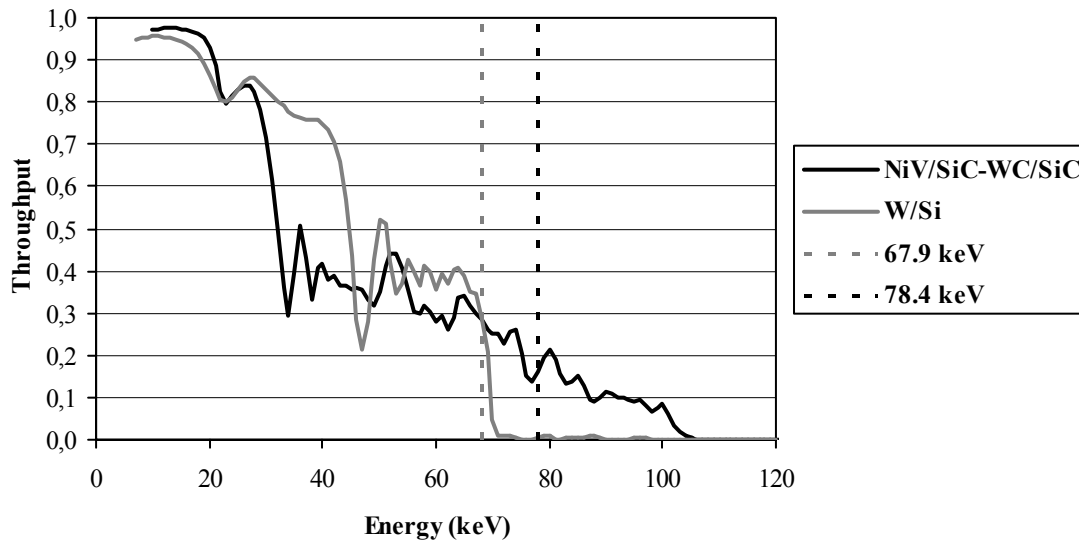


Fig. 6. The throughput after two reflections (reflectivity squared) as function of energy for a NuSTAR W/Si coating and for a NiV/SiC-WC/SiC coating. The designs used for this plot are calculated at an angle of 0.11 deg. The two vertical dotted lines are the two ^{44}Ti lines. The parameters for the two coatings are shown in table 3.

	d_{\min} (nm)	d_{\max} (nm)	N	c	Gamma	Roughness (nm)
W/Si	3.5	26.0	200	0.21	0.40	0.45
Si (substrate)						0.25
NiV/SiC	4.6	40.0	150	0.26	0.50	0.57
WC/SiC	3.2	4.6	90	0.20	0.40	0.22
Si (substrate)						0.25

Table 3. The parameters for the two throughput curves in fig. 6. The designs used for in this table are calculated at an angle of 0.11 deg.

From the parameters in table 3 it can be seen that the numbers of bilayer in the stacked coating is 20 % higher than for the W/Si coating. Also the maximum d-spacing for the NiV/SiC is much higher than for the W/Si coating. One need that since the electron density of NiV is much lower than for WC but since the absorption is much smaller one can benefit from adding the extra layers.

5. CONCLUSION

Both W and Pt, which is the traditional candidates for high Z-materials for hard X-ray telescopes have absorption edges very close to the two interesting ^{44}Ti lines at 67.9 keV and 78.4 keV. We have in this paper discussed the roughness as function of d-spacing for several different high Z-materials combined with SiC. Both Cu/SiC and NiV/SiC that do not have absorption edges at high energy are coating to rough to produce the smallest d-spacings required. By using stacked multilayers, two multilayer with different material combinations on top of each other, one can design coatings that will allow throughput at higher energies than the traditional multilayer. As an example we have coated and characterized a depth graded multilayer of 100[NiV/SiC] / 70[WC/SiC] / Si.

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